FINAL DRAFT

Quality Assurance Project Plan (QAPP) for the Columbia River Pacific Lamprey Toxics Study

Tissue Investigation Willamette Falls, John Day Dam and Sherar's Falls, Oregon

A. Project Management

A1.1 EPA Region 10 – Seattle, WA Approval Page

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A1.3 Oregon DEQ Approval Page

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A4. Project/Task Organization

This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) activities/procedures that will be used while collecting samples for the Columbia River Pacific Lamprey Toxics Study during the 2009 field season. This study will be closely coordinated with the Mid-Columbia Toxics Study, with respect to laboratory analytical procedures. The data for this project will be collected using the "Mid-Columbia Field Methods Manual for probabilistic sampling in Oregon and Washington" (Hayslip and Herger, 2008).

This document covers the probabilistic study design sampling. The project includes the Willamette Falls, Bonneville Dam and Sherars' Falls (Deschutes River) sites. All sites will be sampled between August 1 and August 31, 2009.
This QAPP was prepared according to guidance presented in the document *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5 (USEPA 1999). Reference to the QAPP elements described in the guidance document are included herein.

The project team organization provides the framework for conducting the sample collection task to meet study objectives. The organizational structure and function also facilitate project performance and adherence to QC procedures and QA requirements. Key roles are filled by those persons responsible for ensuring the collection and processing of valid data and for routinely assessing the data for precision and accuracy, as well as the persons responsible for approving and accepting final products and deliverables. The project and QA personnel include staff from USEPA, Columbia River Inter-Tribal Fish Commission, Oregon Department of Human Services and Oregon Department of Environmental Quality (ODEQ).

The CRITFC Project Manager, Bob Heinith, will supervise staff, coordinate and participate in sampling, secure permits if any, assure laboratory work is completed on time and review completion reports.

The Oregon Human Services Managers, David Farrer and Barbara Stifel will perform the human health risk assessment.and prepare a report.

The **EPA Project Managers** (Kris Carrie and Gina Grepo-Grove) will supervise staff, oversee study design and site selection, and ensure adherence to design objectives. Managers also review and approve the project work plan, QAPP, and other materials developed to support the project.

The **USEPA Quality Assurance Manager,** Don Matheny and Bob Ozretich, and the **Oregon DEQ QA Officer**, Chris Redman, will be responsible for reviewing and approving all Quality Assurance Project Plans (QAPPs).

The **Oregon Field Project Sampling Coordinator / ODEQ Project Manager**, Larry Caton and Aaron Borisheko will ensure that QA/QC protocols are maintained throughout the sample collection and preparation processes in 2009. Evaluations will include reviewing all required documentation for completeness and seeing that any problems encountered outside normal operating conditions are documented and addressed, and verifying all other QA/QC procedures identified in the QAPP are followed.

Field Sampling Teams:

CRITFC and tribal field staff, are responsible for performing the field work, including collection, preparation, and shipment of samples and completion of field sampling records. The Field Sampling Teams will include scientific staff with specialization and technical competence in field sampling activities to effectively and efficiently perform the required work. They must perform all work in adherence with the project work plan and QAPP. In this role, Field Sampling Teams are responsible for:

• receiving and inspecting the sample containers,
• completing and signing appropriate field records,
• assigning tracking numbers to each sample,
• verifying the completeness and accuracy of chain-of-custody documentation,
• controlling and monitoring access to samples while in their custody, and
• initiating shipment of the samples to appropriate destinations.

Sample Shipping Address

Oregon DEQ Laboratory
3150 NW 229th Ave., Suite 150
Hillsboro, OR 97124
Phone: (503) 693-5700 Fax: (503) 693-4999

Contact: **Heather Cayton (503) 693-5773**

A5. Problem Definition/Background

Improving water quality and assessing effects of contaminants on biota in the Columbia River Basin has been a priority for States, Tribes, Federal Agencies and others for many years. The Basin was identified by EPA as one of seven Great Water Bodies in EPA's 2006-2011 Strategic Plan (U.S. EPA 2006). The goal of EPA's Strategic Plan for the Columbia Basin is to prevent water pollution, and improve and protect water quality and ecosystems to reduce risks to human health and the environment.

EPA studies and state monitoring programs have found significant levels of toxicants in fish and the water of the Columbia River. Accumulation of toxicants in fish threatens the survival of fish species, and human consumption of these fish can lead to health problems. Many governments, communities and citizens have rallied to launch long term and intensive recovery efforts to restore fish health and populations in the Columbia River.

Contaminants, such as polychlorinated biphenyls (PCBs) and mercury, have been found in various fish species in rivers throughout the Columbia River Basin. To ensure the continued good health of the citizens of the Columbia River Basin, the states issue fish consumption advisories for specific fish species in water bodies that exceed human health criteria as identified by Oregon Department of Human Services and the Washington Deparatment of Health. Fish consumption advisories may be issued to protect the general public or sensitive populations such as women of childbearing age, nursing mothers, pregnant women, and children.

The health implications from ingesting Willamette River Pacific lamprey containing toxic accumulations has been studied. In 2004, the Siletz Tribe, through a EPA grant, requested that the Oregon Department of Human Services (ODHS) through the Superfund Health Investigation and Education Program investigate the risks to tribal members from ingesting lamprey collected at Willamette Falls (Siletz 2004; Stone 2003). Samples were taken, preserved on dry ice and shipped to a laboratory for analysis. Several pollutants were identified in the samples, with levels of mercury, DDT, Chlordane, Dieldrin and PCBS considered a health risk, particularly to pregnant women and children.

To tribal members, Pacific lamprey, or “eels” are just as important culturally and spiritually as salmon. Traditionally, while salmon were only available for sustenance during certain periods, lamprey were always present and provided an important backstop in lean times. For various reasons, which may include poor passage at mainstem and tributary dams, loss of habitat, poor water quality and increased marine mammal, bird and fish predation, lamprey are quickly disappearing from most of the Columbia and Snake Rivers. It is not know at this time whether toxicants in the lamprey environment may be a limiting factor to lamprey populations. To address the severe Pacific lamprey losses in the Columbia River Basins, CRITFC and its member tribes developed a “Tribal Pacific Lamprey Restoration Plan for the Columbia River Basin” (CRITFC 2008). A key objective of the Plan is to evaluate toxic pollutant levels in lamprey throughout the basin, assess the health impact of ingestion of lamprey, and take actions to reduce toxic pollutants in lamprey.

We propose to obtain a total of 7 adult lamprey composite samples from Willamette Falls (2 samples), John Day Dam (3 samples) and Sherars’ Falls on the Deschutes River (1 samples), of 5-7 lamprey in each composite sample. These composite samples will be analyzed for the key toxic contaminants targeted in the 2009-2010 Mid-Columbia QAPP and past studies by ODEQ in association with the 2009-2010 Mid-Columbia Toxics Study. In turn, these analyses will assist in: 1) deriving potential health implications from ingestion of these animals and, 2) assessing the potential ecological effects of these toxics on lamprey.

Tribal biologists from CRITFC and the Confederated Tribes of the Warm Springs and the Confederated Tribes of the Umatilla Indian Reservation will be involved in obtaining composite samples. The Oregon Department of Human Services will collaborate with ODEQ in reviewing the sampling design and on specific issues related to contaminants.

The Columbia River is water quality limited for DDT, DDE, PCBs, arsenic, and PAHs. The states, tribes and federal government and non-governmental organizations in the Columbia Basin are all engaged in efforts to restore and improve the water, land and air quality of the Columbia River Basin. They have committed to work together to restore critical ecosystems. The Columbia River Toxics Reduction Working Group, a multi-entity group lead by EPA, helped determine the list of contaminants that will be sampled for this project.

A6. Project/Task Description

This Columbia River Pacific Lamprey Toxics Study will be closely coordinated with the Mid-Columbia River Toxics Study which is evaluating ecological conditions and contaminant sources that build upon previous Environmental Monitoring and Assessment Program (EMAP) studies conducted in the Lower Columbia River, from Bonneville Dam to the river mouth. This project covers three sites within the basin, Willamette Falls, John Day Dam and Sherars Falls on the Deschutes River, Oregon.

The data collected will be used to assess health implications and ecological effects of toxic contaminants in Pacific Lamprey. The data will be used to evaluate the water quality of the river from an ecological and Clean Water Act perspective. Table 2 provides an overview of the types of samples that will be collected for this study. The study will assess the condition of ecological resources and identify stressors associated with degradation (e.g. 305b reporting).

Table 1. Sample Matrices and Parameters

Parameter	Matrix	Method	Sample Year(s)
PCB Congeners	Whole-body lamprey composites (5-7 lamprey/sample) for ecological and human health endpoints	Hand or dip net collection by CRITFC or other Tribal harvesters; ecological and human health analyses at Oregon DEQ lab	2009
Chlorinated Pesticides	Whole-body lamprey composites (5-7 lamprey/sample) for ecological and human health endpoints	Hand or dip net collection by CRITFC or other Tribal harvesters; ecological and human health analyses at Oregon DEQ lab	2009
DDT & Metabolites	Whole-body lamprey composites (5-7 lamprey/sample) for ecological and human health endpoints	Hand or dip net collection by CRITFC or other Tribal harvesters; ecological and human health analyses at Oregon DEQ lab	2009
PBDE Congeners	Whole-body lamprey composites (5-7 lamprey/sample) for ecological and human health end points	Hand or dip net collection by CRITFC or other Tribal harvesters; ecological and human health analyses at Oregon DEQ lab	2009
Mercury	Whole-body lamprey composites (5-7 lamprey/sample) for ecological and human health endpoints	Hand or dip net collection by CRITFC or other Tribal harvesters; ecological and human health analyses at Oregon DEQ lab	2009

CRITFC's member tribes, EPA Region 10, ODEQ, ODHS, ODFW and other and local decision makers need additional water quality, biological and habitat data to complete a mid-Columbia ecological condition assessment, and a contaminant source assessment. The main questions for this particular study are:

- Do priority contaminants in Pacific Lamprey identified by the EPA's Columbia River Toxics Reduction Working Group pose a health or ecological risk?
- What are contaminant levels in whole body adult Pacific lamprey?
- What are the potential ecological issues for lamprey that contain these contaminant levels?

A7. Quality Objectives and Criteria for Measurement Data

Measurement Performance Criteria

Measurement performance criteria are quantitative statistics that are used to interpret the degree of acceptability or utility of the data to the user. These criteria, also known as data quality indicators (DQIs), include the following: precision, accuracy, representativeness, completeness, and comparability.

Precision

Precision is a measure of internal method consistency. It is demonstrated by the degree of agreement between individual measurements (or values) of the same property of a sample, measured under similar conditions.

Accuracy

Accuracy is defined as the degree of agreement between an observed value and an accepted reference or true value. Accuracy is a combination of random error (precision) and systematic error (bias), introduced during sampling and analytical operations. Bias is the systematic distortion of a measurement process that causes errors in one direction, so that the expected sample measurement is always greater or lesser to the same degree than the sample's true value.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter, and variations at a sampling point, a process condition, or an environmental condition.

Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid according to specific criteria and entered into the data management system. To optimize completeness, every Effort is made to avoid sample and/or data loss. Accidents during sample transport or lab activities that cause the loss of the original samples will result in irreparable loss of data, which will reduce the ability to perform analyses, integrate results, and prepare reports. Samples will be stored and transported in unbreakable (plastic) containers (i.e., insulated ice chests). The project manager will decide whether to analyze samples that fail holding time or preservation requirements, and how to flag any related data.

On rare occasions, laboratories lose or compromise samples (breaking jars, etc.). The project manger will decide if these samples are salvageable and worth analyzing, and how to flag any related data.

Completeness, in the case of this project, is the number of valid samples collected relative to the number of samples that are planned to be collected. The completeness goal for this project is 90%.

Comparability

Comparability is an expression of the confidence with which one data set can be compared with another data set. Comparability is dependent on the proper design of the sampling program and on adherence to accepted sampling techniques, standard operating procedures, and quality assurance guidelines. Comparability of data will be accomplished by standardizing the sampling season, the field sampling methods, and the field training as follows: all samples will be collected during the summer (July - August) and all samples will be collected and prepared for shipment according to standard operating procedures contained in this QAPP.

Table 2. Data Quality Indicators for Fish Tissue – Ecological and Human Health Endpoints.

(All 2009 samples will be analyzed at the Oregon DEQ Lab.)

Matrix: Whole lamprey	Analytical Method	Matrix Target L.O.D.	Matrix Target L.O.Q.	Precision RPD	Matrix Spike Recovery	Lab Control Standard	Surrogates	Standard Reference Material
Mercury	EPA 7473	0.01 ug/g-wet	0.05 ug/g-wet	< 30% RPD	70 - 130% Control Chart Limits: Warning: 2 std. dev. Control: 3 std. dev.	+/-30% Control Chart Limits: Warning: 2 std. dev. Control: 3 std. dev.	N.A.	True Value Limits: Warning: 95% C.I. Control: +/-10% of 95% C.I.
Lipids	Gravimetric	0.02%	1%	< 30% RPD	N.A.	N.A.	N.A.	N.A.
Solids	Gravimetric	0.02%	1%	< 30% RPD	N.A.	N.A.	N.A.	N.A.
PCB	EPA 8270	1 ng/g-wet	10 ng/g-wet	≤ 30% RPD	50 - 120% Control Chart Limits: Warning: 2 std. dev. Control: 3 std. dev.	50 - 120% Control Chart Limits: Warning: 2 std. dev. Control: 3 std. dev.	30 - 150% Control Chart Limits: Warning: 2 std. dev. Control: 3 std. dev.	True Value* Limits: Warning: +/- 20% of 95% C.I. Control: +/-30% of 95% C.I.
PBDE Pesticides & DDTs	Same as for PCB							

*Marginal Exceedances allowed, per NELAC Appendix D.1.1.2.1e. Once the lab has 20 SRM replicates, QC limits will be based on control chart standard deviation.

A8. Special Training Requirements/Certification

Each Field Sampling Team is required to have the necessary knowledge and experience to perform all field activities. This includes skills in fish collection (especially EPA Method 1669).. Sampling sites will be foot accessible. It also includes training in project-specific sample collection and handling procedures. The field sampling crews will be comprised fisheries biologists with a strong technical background in fisheries and water quality sampling activities. Each Field Sampling Team will consist of (at a minimum[1]) one experienced fisheries biologist..

This QAPP, the field methods manual, and orientation materials will be distributed to all sampling personnel. Project orientation sessions will be set up to distribute and discuss training materials. Materials will include detailed instructions for each field procedure. The focus of the orientation will be on sample collection methods, specific details of sample preparation, and strict adherence to the study's protocols.

A9. Documentation and Records

The minimum required data to be recorded for each method is identified in the Field Methods Manual (Hayslip and Herger, 2008), and the SPMD deployment SOP. Thorough documentation of all field sample collection and handling activities is necessary for proper processing in the laboratory and, ultimately, for the interpretation of study results. Field sample collection and handling will be documented (for each sampling site) using the following forms:

- Sample Custody and Analysis Required Form (s)
- Fish Tissue Collection Form

The above forms will be completed as described in the Field Methods Manual (Hayslip and Herger, 2008). All label entries will ideally be made in indelible ink and will be consistent with sample information on the appropriate field forms. (When weather or boating conditions prevent the use of indelible ink, pencil is an acceptable alternative.)

Samples will be hand delivered or to the sample preparation laboratory to meet sample holding time or ensure preservation. Whole fish will be placed in appropriate containers in dry ice or frozen and delivered to the ODEQ lab for analyses.

If any change(s) in this QAPP is (are) required during the study, a memo will be sent to each person on the distribution list describing the change(s), following approval by the Project Manager. Any and all memos announcing changes must be attached to the QAPP.

All documents and records prepared for this project will be maintained by CRITFC and ODEQ during the project, and retained for a period of two years following completion of the project.

[1] ODEQ may reduce field crew requirements to one operator and one field technician for some project phases, especially when two boat crews are working together.

B. Data Acquisition

B1. Sampling Process Design

The objective of the Columbia River Pacific Lamprey Toxics Study is to assess toxic levels in Pacific lamprey collected at three comparative sites using the field methods described in the Mid-Columbia Toxics Study (2009-2010 Mid-Columbia Toxics Study). The data will be used to understand the potential effects of toxics in Pacific lamprey from an ecological and health perspective.

Sampling Period

Field sampling will be conducted during the period when water and weather conditions are conducive to safe and efficient field sampling. The 2009 index period sampling will be from August 1- August 31.

Sample Frame

For the purposes of this study, the target population will be the Columbia River at John Day Dam, the Willamette River at Willamette Falls and Sherar's Falls on the Deschutes River, Oregon.

Selection of Sites for Sampling

The three sites identified above in the sample frame were chosen due to the ability to acquire lamprey at them and to provide data from a spectrum of the lower Columbia River and tributaries. The depressed status of lamprey abundance in 2009 limited collection to these sites.

B2. Sampling Methods

Most sampling methods for this project are fully described in the "Mid-Columbia Field Methods Manual for probabilistic sampling in Oregon and Washington" (Hayslip and Herger, 2008). The "Clean Hands/Dirty Hands" procedure for trace metal sampling is fully described in EPA Method 1669. The following is a summary of those more detailed methods.

Field Collection Methods

Sampling methods shall include: hand collection in special lamprey traps and dip netting. Collection of fish by any technique will be designated by the stipulations of the federal, state and tribal permits, if any. Copies of the permits will be in the possession of the field sampler at all times.

The CRITFC, ODEQ and ODHS field coordinators shall ensure that the necessary safety equipment and emergency information are always available. The methods of sample collection that will be used for this project are discussed in the following paragraphs.

Dip nets or hand and trap collection will be used for 1) collecting adult lamprey in the fish ladder at Willamette Falls and/or at the Falls directly from tribal fishers., 2) collecting lamprey from the John Day Dam fishway and 3) collecting samples at Sherars' Falls on the Deschutes River, Oregon. Once a fish is caught, the dip net will be pulled to the surface and the fish removed. Only the fish selected for this project will be kept..

B3. Sample Handling and Custody Requirements

Primary concern with sample handling and processing is to avoid sources of extraneous tissue contamination including contamination from sampling gear, spilled engine fuel (gasoline or diesel), engine exhaust, dust, ice chests, and ice used for cooling. All potential sources of contamination in the field should be identified and appropriate steps taken to minimize or eliminate them. Ice chests should be scrubbed clean with detergent and rinsed with distilled water after each use to prevent contamination. *To avoid contamination from melting ice, samples should be placed in waterproof plastic bags.* Sampling equipment that has obviously been contaminated by oils, grease, diesel fuel, or gasoline should not be used. All utensils or equipment that will be used directly in handling fish (e.g., fish measuring board, scales) should be cleaned in the laboratory prior to each sampling trip, rinsed in acetone and pesticide-grade hexane, and stored in aluminum foil until use. Between sampling sites, the field collection team should clean each measurement device by rinsing it with ambient water and rewrapping it in aluminum foil to prevent contamination. Likewise, the loss of contaminants can be prevented in the field by ensuring that the sample collected remains intact, i.e., sample collection procedures should be performed with the intention of minimizing the laceration of fish skin.

Individuals of the selected target species will be rinsed in ambient water to remove any foreign material from the external surface. A nine-character composite sample identification number consisting of the two-character state abbreviation, two-number year abbreviation, 3-digit site identification number, composite type (“H” or “E” for human health or ecological endpoint species), and sample type (“S” or “D” for standard or duplicate) will be assigned by the field teams for each composite collected. The composite sample specimen number and information regarding the fish specimens will be recorded on the field record forms.

A Chain-of-Custody Form acts as a record of sample shipment and a catalog of the contents of each shipment (coinciding with information on the field record). Because the information needed is somewhat redundant with the field data, we will use a single form to record the field data and to serve as the chain-of-custody form as well. Entries will be made in ink. One Xeroxed copy is retained by the sampler and the original is sealed inside the shipping container. The information on the field data form is in the previous section. Prior to shipping the bottom of the form is completed for the Chain of Custody. This requires the signature of the person relinquishing the sample, the date, and the time.

Upon receipt of the samples, the analytical Laboratory will record the arrival time on the chain of custody form. Any observations regarding the shipment (e.g., torn or damaged packaging, insufficient dry ice) also will be documented on the chain of custody form.

B4. Analytical Methods Requirements

The list of laboratory analytes are shown in Tables 3 and 4. The ODEQ laboratory will use EPA or other standard methods which have proven effective in water or tissue matrices. Proposed analytical methods can achieve the detections limits and other quality controls required to meet project objectives listed in Tables 5 through 9. The laboratories may use other suitable methods, provided that performance based measures are achieved. These quality control measures include the use of standard reference materials (SRM), laboratory control standards (LCS), matrix spikes and matrix spike duplicates (MS/MSD), continuing calibration verification (CCV), surrogates, internal standards, laboratory blanks, replicate analyses, and other method specific quality control activities.

Ideally, all laboratory analytical results will be reported to the laboratory's limit of detection (LOD) *when a measurable signal is observed*. Result *values* between the laboratory's LOD and limit of quantification (LOQ) *will be reported and flagged* as "Less than Limit of Quantification" (<LOQ). *When no measurable signal is observed*, the result will be reported as the *LOQ value* and flagged with a "less than" symbol (<). These reporting procedures are intended to reduce the amount of censored data, facilitate statistical analyses, and permit qualitative assessment of blank contamination.

Fish Tissue

The DEQ laboratory will analyze the four ecological endpoint and health endpoint for lamprey samples. Each sample will be weighed and measured for length prior to being frozen whole in the field.

Whole samples will be homogenized at the DEQ laboratory according to ODEQ's fish homogenization SOP, which complies with EPA's National Fish Health Advisory Laboratory Procedures, Section 7 (EPA 2000)) [2] The work area will be fitted with a glass bench liner, and glass or foil-covered cutting boards will be used. Ceramic knives will be used to cube partially frozen tissue prior to homogenization in a Buchi blender with ceramic blades. Homogenates will be placed on cleaned foil sheets, and mixed as described in EPA's SOP, transferred to trace-cleaned muffled jars with Teflon-coated utensils, and frozen at -20 C.

DEQ will use appropriate analytical methods to achieve the required measurement quality objectives. In 2009, DEQ acquired a high resolution GCMS and will likely generate data that exceeds the project's measurement quality objectives. The new instrument will significantly improve DEQ's analytical capabilities by reducing matrix interferences, and improving sensitivity. (Analytical methods will be changed to reflect the new instrumentation, but the project's data quality objectives have not changed).

Table 3. Target Fish Tissue Analytes for Ecological Endpoints and Human Health Endpoints Showing Required Detection Limits.

BZ #	Analyte name	Ecological Endpoints (ODEQ)	Human Health Endpoints (ODEQ)	Detection Limits
PCB Congeners (via Accelerated Solvent Extractions/Solvent Cleanup/Lipid partitioning/ Electron Capture Methods)				
8	2,4-Dichlorobiphenyl, #8 (34883-43-7)	Yes	Yes	0.625 µg/Kg
18	2,2',5-Trichlorobiphenyl, #18 (37680-65-2)	Yes	Yes	
28	2,4,4'-Trichlorobiphenyl, #28 (7012-37-5)	Yes	Yes	
44	2,2',3,5'-Tetrachlorobiphenyl, #44 (41464-39-5)	Yes	Yes	
52	2,2',5,5'-Tetrachlorobiphenyl, #52 (35693-99-3)	Yes	Yes	
66	2,3',4,4'-Tetrachlorobiphenyl, #66 (32598-10-0)	Yes	Yes	
77	3,3',4,4' Tetrachlorobiphenyl, #77* (32598-13-3)	Yes	Yes	
81	3,4,4,5- Tetrachlorobiphenyl, #81 (70362-50-4)	Yes		
101	2,2',4,5,5'-Pentachlorobiphenyl, #101 (37680-73-2)	Yes	Yes	
105	2,3,3',4,4'-Pentachlorobiphenyl, #105 (32598-14-4)	Yes	Yes	
110	2,3,3',4',6-pentachlorobiphenyl	**Yes**	Yes	

[2] Exceptions to Section 7 procedures include removal of the belly flap from fish fillets; metal instruments and utensils not being cleaned with acid; and implements rinsed with a 1:1 acetone:hexane mixture rather than isopropanol or acetone alone.

BZ #	Analyte name	Ecological Endpoints (ODEQ)	Human Health Endpoints (ODEQ)	Detection Limits
118	2,3',4,4',5-Pentachlorobiphenyl, #118 (31508-00-6)	Yes	Yes	
126	3,3',4,4',5 Pentachlorobiphenyl, #126	Yes	Yes	
128	2,2',3,3',4,4'-Hexachlorobiphenyl, #128 (38380-07-3)	Yes	Yes	
138	2,2',3,4,4',5-Hexachlorobiphenyl, #138 (35065-28-2)	Yes	Yes	
153	2,2',4,4',5,5'-Hexachlorobiphenyl, #153 (35065-27-1)	Yes	Yes	
169	3,3',4,4',5,5' Hexachlorobiphenyl, #169 (32774-16-6)	Yes		
170	2,2',3,3',4,4',5-Heptachlorobiphenyl, #170 (35065-30-6)	Yes	Yes	
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl, #180 (35065-29-3)	Yes	Yes	
187	2,2',3,4',5,5',6-Heptachlorobiphenyl, #187 (52663-68-0)	Yes	Yes	
195	2,2',3,3',4,4',5,6-Octachlorobiphenyl, #195 (52663-78-2)	Yes	Yes	
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl, #206 (40186-72-9)	Yes	Yes	
209	Decachlorobiphenyl, #209 (2051-24-3)	Yes	Yes	
Chlorinated Pesticides (via Accelerated Solvent Extraction/Solvent Cleanup/Electron Capture Methods)				
	Aldrin (309-00-2)	Yes	Yes	Detection limit (ppb) µg/Kg wet weight approximately 0.12 - 0.73
	Alpha-Chlordane		Yes	
	Alpha-BHC	Yes	Yes	
	Chlordane-cis (5103-71-9)	Yes	Yes	
	Chlordane-trans (5103-74-2)	Yes	Yes	
	Dieldrin (60-57-1)	Yes	Yes	
	Endosulfan I (959-98-8)	Yes	Yes	
	Endosulfan II (33213-65-9)	Yes	Yes	
	Endosulfan sulfate			
	Endrin (72-20-8)	Yes	Yes	
	Heptachlor (76-44-8)	Yes	Yes	
	Heptachlor Epoxide (1024-57-3)	Yes	Yes	
	Hexachlorobenzene (118-74-1)	Yes	Yes	
	Hexachlorocyclohexane [Gamma-HC/Lindane] (58-89-87)	Yes	Yes	
	Mirex (2385-85-5)	Yes	Yes	
	trans-Nonachlor (3765-80-5)	Yes	Yes	
	cis-Nonachlor (5103-73-1)	Yes	Yes	
	Oxychlordane (27304-13-8)	Yes	Yes	
DDT & Metabolites (via Accelerated Solvent Extraction/Solvent Cleanup/Electron Capture Methods)				
	2,4'-DDD (53-19-0)	Yes	Yes	Detection limit (ppb) µg/Kg wet weight approximately 0.12 - 0.73
	4,4'-DDD (72-54-8)	Yes	Yes	
	2,4'-DDE (3424-82-6)	Yes	Yes	
	4,4'-DDE (72-55-9)	Yes	Yes	
	2,4'-DDT (789-02-6)	Yes	Yes	
	4,4'-DDT (50-29-3)	Yes	Yes	
PBDE Congeners (via Accelerated Solvent Extraction/Solvent Cleanup/Electron Capture Methods)				
28	2,4,4’-Tribromodiphenyl ether	Yes		0.625 µg/Kg
47	2,2’,4,4’-Tetrabromodiphenyl ether	Yes	Yes	
66	2,3’,4,4’-Tetrabromodiphenyl ether	Yes		
85	2,2’,3,4,4’-Pentabromodiphenyl ether	Yes		
99	2,2’,4,4’,5-Pentabromodiphenyl ether	Yes	Yes	
100	2,2’,4,4’,6-Pentabromodiphenyl ether	Yes	Yes	
138	2,2’,3,4,4’,5’-Hexabromodiphenyl ether	Yes		
153	2,2’,4,4’,5,5’-Hexabromodiphenyl ether	Yes	Yes	

BZ #	Analyte name	Ecological Endpoints (ODEQ)	Human Health Endpoints (ODEQ)	Detection Limits
154	2,2’,4,4’,5,6’-Hexabromodiphenyl ether	Yes	Yes	
183	2,2',3,4,4',5',6-Heptabromodiphenyl ether	Yes	Yes	
209	Decabromodiphenyl ether	Yes		
Metals (via gold amalgamation)				
	Mercury (7439-97-6) (via ICP Methods)	Yes	Yes	0.01 (ppm) ug/g wet weight
Additional Measurements				
	Percent Moisture (Karl-Fisher Titration) or Percent Solids	Yes	Yes	
	Lipids (Gravimeteric Method)	Yes	Yes	

Note: Since Table 3 was prepared, ODEQ has acquired a high resolution GCMS, and expects to report more analytes than originally requested with improved reporting limits, as per EPA Methods 1613, 1614, 1668, and 1699. Analytes not originally included in Table 3 will be flagged according to the established data quality indicators for the analyte group (e.g. PCBs).

Table 4. Target SPMD Analytes Showing Target Detection Limits.

BZ #	Analyte name	Detection Limits
	PCB Congeners (HRMS Methods)	
8	2,4-Dichlorobiphenyl, #8 (34883-43-7)	0.5 ng/membrane
18	2,2',5-Trichlorobiphenyl, #18 (37680-65-2)	
28	2,4,4'-Trichlorobiphenyl, #28 (7012-37-5)	
44	2,2',3,5'-Tetrachlorobiphenyl, #44 (41464-39-5)	
52	2,2',5,5'-Tetrachlorobiphenyl, #52 (35693-99-3)	
66	2,3',4,4'-Tetrachlorobiphenyl, #66 (32598-10-0)	
77	3,3',4,4' Tetrachlorobiphenyl, #77* (32598-13-3)	
81	3,4,4,5- Tetrachlorobiphenyl, #81 (70362-50-4)	
101	2,2',4,5,5'-Pentachlorobiphenyl, #101 (37680-73-2)	
105	2,3,3',4,4'-Pentachlorobiphenyl, #105 (32598-14-4)	
110	2,3,3',4',6-pentachlorobiphenyl	
118	2,3',4,4',5-Pentachlorobiphenyl, #118 (31508-00-6)	
126	3,3',4,4',5 Pentachlorobiphenyl, #126	
128	2,2',3,3',4,4'-Hexachlorobiphenyl, #128 (38380-07-3)	
138	2,2',3,4,4',5-Hexachlorobiphenyl, #138 (35065-28-2)	
153	2,2',4,4',5,5'-Hexachlorobiphenyl, #153 (35065-27-1)	
169	3,3',4,4',5,5' Hexachlorobiphenyl, #169 (32774-16-6)	
170	2,2',3,3',4,4',5-Heptachlorobiphenyl, #170 (35065-30-6)	
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl, #180 (35065-29-3)	
187	2,2',3,4',5,5',6-Heptachlorobiphenyl, #187 (52663-68-0)	
195	2,2',3,3',4,4',5,6-Octachlorobiphenyl, #195 (52663-78-2)	
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl, #206 (40186-72-9)	
209	Decachlorobiphenyl, #209 (2051-24-3)	
	Chlorinated Pesticides (HRMS Methods)	
	Aldrin (309-00-2)	0.5 ng/membrane
	Alpha-Chlordane	
	Alpha-BHC	
	Chlordane-cis (5103-71-9)	
	Chlordane-trans (5103-74-2)	
	Dieldrin (60-57-1)	
	Endosulfan I (959-98-8)	
	Endosulfan II (33213-65-9)	
	Endosulfan sulfate	
	Endrin (72-20-8)	
	Heptachlor (76-44-8)	
	Heptachlor Epoxide (1024-57-3)	
	Hexachlorobenzene (118-74-1)	
	Hexachlorocyclohexane [Gamma-HC/Lindane] (58-89-87)	
	Mirex (2385-85-5)	
	trans-Nonachlor (3765-80-5)	
	cis-Nonachlor (5103-73-1)	
	Oxychlordane (27304-13-8)	
	DDT & Metabolites (HRMS Methods)	

BZ #	Analyte name	Detection Limits
	2,4'-DDD (53-19-0)	0.5 ng/membrane
	4,4'-DDD (72-54-8)	
	2,4'-DDE (3424-82-6)	
	4,4'-DDE (72-55-9)	
	2,4'-DDT (789-02-6)	
	4,4'-DDT (50-29-3)	
	PBDE Congeners (HRMS Methods)	
28	2,4,4'-Tribromodiphenyl ether	0.5 ng/membrane
47	2,2',4,4'-Tetrabromodiphenyl ether	
66	2,3',4,4'-Tetrabromodiphenyl ether	
85	2,2',3,4,4'-Pentabromodiphenyl ether	
99	2,2',4,4',5-Pentabromodiphenyl ether	
100	2,2',4,4',6-Pentabromodiphenyl ether	
138	2,2',3,4,4',5'-Hexabromodiphenyl ether	
153	2,2',4,4',5,5'-Hexabromodiphenyl ether	
154	2,2',4,4',5,6'-Hexabromodiphenyl ether	
183	2,2',3,4,4',5',6-Heptabromodiphenyl ether	
209	Decabromodiphenyl ether	
	PAHs (GCMS Methods)	
	Anthracene	1.0 ug/membrane
	Benz(a)anthracene	
	Dibenz(a,h)anthracene	
	Biphenyl	
	Chrysene	
	Fluoranthene	
	Benzo(b)fluoranthene	
	Benzo(k)fluoranthene	
	Fluorene	
	Acenaphthene	
	Naphthalene	
	Acenaphthylene	
	1-methylnaphthalene	
	2-methylnaphthalene	
	2,6-dimethylnaphthalene	
	2,3,5-trimethylnaphthalene	
	Benzo(g,h,i)perylene	
	Phenanthrene	
	1-methylphenanthrene	
	Pyrene	
	Benzo(a)pyrene	
	Indeno(1,2,3-c,d)pyrene	
	Dibenzothiophene	
	Phenanthrene	

B5. Quality Control Requirements

Data quality is addressed, in part, by consistent performance of valid procedures documented in the Mid-Columbia Toxics Study. All laboratory contaminant analyses performed in this study will be performed in conjunction with the standard QC elements (see Tables 5 and 6). Additional QC requirements are in Table 17 and 18 below.

Table 5. Additional QC Requirements

Parameter	QC	Frequency	Required Parameters	Corrective Action
Percent Moisture	QC Std, (Karl Fisher only)	Used to validate instrument	Average of 3 per batch	±10% Recovery
	Duplicate	Used to determine precision	1per batch	≤ 10% RPD
Percent Lipid	Duplicate	Used to determine precision	1per batch	≤ 20% RPD

Table 6. Additional QC Specifications for Ecological Endpoint Fish Tissue Samples – Mercury

QA/QC Sample or Element	Control Limit	Frequency
QC Blank	< 5 ppb	one per batch at the beginning of run
Standard Reference Material (CRC DORM-2)	80-120% Recovery (average of 2 runs)	one per batch prior to sample analysis
QC Calibration Check Standard (secondary source)	80-120% Recovery	twice at start of batch (one for low curve and one for high curve) and once at end
Matrix Spike/Standard Addition	80-120% Recovery (average of 2 runs)	one per batch at the beginning

B6. Instrument/Equipment Testing, Inspection, and Maintenance Requirements

All field equipment will be inspected prior to sampling activities to ensure that proper use requirements are met (e.g., boats or are operating correctly, nets are without defects). Inspection of field equipment will occur well in advance of the field operation to allow time for replacement or repair of defective equipment, and the field team will be equipped with proper backup equipment to prevent lost time on site. One member of each field team should gather and inspect all equipment on the equipment and supply list prior to each sampling event.

B7. Instrument Calibration and Frequency

All meters used by field teams will be calibrated according to the manufacturer's operating Instructions on a daily basis, while in use.

B8. Inspection/Acceptance Requirements for Supplies and Consumables

Careful and thorough planning is necessary to ensure the efficient and effective completion of the field sample collection task. A general checklist of field equipment and supplies is provided in the Field Methods Manual (Hayslip and Herger, 2008). It will be the responsibility of each field team to gather and inspect the necessary sampling gear prior to the sampling event and to inspect the sample Packaging and shipping supplies. Defective packaging and shipping supplies (e.g., torn or damaged polyethylene sample tubing) will be discarded.

B9. Data Acquisition Requirements (Non-direct Measurements)

Non-direct measurements will include identification and/or verification of each sample site location (i.e., latitude and longitude). Columbia River flow data may be obtained from state or federal agency gages to complement data analysis and reporting.

B10. Data Management

Field Data

Monitoring personnel shall collect and report data on the datasheets provided for this project in the field methods manual (Hayslip and Herger, 2008). The data sheets will be kept and maintained in an organized file. Field datasheets and other sample documentation shall be initially reviewed for transcription errors, precision, completeness, anomalous data, and other general problems.

Samples will be documented and tracked via Sample Identification Labels, Field Record Forms, and Sample Custody & Analysis Required Forms. Field team leaders will be responsible for reviewing all completed field forms. Any corrections should be noted, initialed, and dated by the reviewer.
Field, COC, and Analysis Required Forms received at ODEQ are electronically scanned and stored in the Laboratory Information Management System (LIMS). Data clerks enter field data into LIMS, and field crews review and validate data in the system.

Laboratory Analytical Data

Composited whole lamprey samples will be analyzed at the ODEQ Lab.
At the DEQ laboratory the sample custodian receives samples, checks and records ice chest temperature, logs the samples into their information management system, and assigns analyses according to the QAPP.

For routine samples analyzed at ODEQ, chemists enter analytical results into LIMS and their work is reviewed by lead chemists and the analytical section manager. The data passes to the Quality Assurance section for review in LIMS. At this point, QA approved data normally moves to laboratory administrator review. For this project, the data will first be reviewed by the ODEQ Field Project Sampling Coordinator, and then by the ODEQ Project Manager. Final analytical reports are generated by the system, and electronic reports are emailed to the Project Manager. Hard copy reports are printed, held for the required time period, and then archived off site. Routine finalized data is automatically transferred from LIMS to the ODEQ LASAR database. (The LASAR database may be queried from ODEQ's website: http://www.deq.state.or.us/lab/lasar.htm). Metadata is available to EPA and others by contacting the ODEQ laboratory's Technical Services Section or the ODEQ Project Manager. Data in LASAR is linked to the Pacific Northwest Water Quality Data Exchange, and will eventually be connected to EPA's Water Quality Exchange.

This data will be stored in the Access database described above in Section B10 "Field Data". The ODEQ Project Manager will work with the various laboratory contacts identified in Table 1, and with the ODEQ Technical Services Section to format data for loading to LASAR.

C. Assessment/Oversight

C1. Assessment and Response Actions

Assessment activities and corrective response actions have been identified to ensure that sample Collection activities are conducted as prescribed and that the measurement quality objectives and data quality objectives established by USEPA are met. The essential steps are as follows:

- identify and define the problem,
- assign responsibility for investigating the problem,
- investigate and determine the cause of the problem,
- assign and accept responsibility for implementing appropriate corrective action,
- establish effectiveness of and implement the corrective action, and
- verify that the corrective action has eliminated the problem.

Immediate corrective actions form part of normal operating procedures and are noted on project Field Record Forms. Problems not solved this way require more formalized, long-term corrective action.

C2. Reports to Management

Annual reports will be produced by ODEQ in the fall of each year and will describe activities during the previous calendar year. These reports will consist of information on project status, highlights, results of QC audits and internal assessments. The project personnel are responsible for report production and distribution.

D. Data Validation and Usability

D1. Data Review, Validation, and Verification Requirements

Data validation and review services provide a method for determining the usability and limitations of data, and provide a standardized data quality assessment. All Field Record Forms and Chain-of-Custody records will be reviewed by the field sampling team for completeness and correctness. Data quality will be assessed by comparing entered data to original data or by comparing results with the measurement performance criteria summarized in tables three through nine to determine whether to accept, reject, or qualify the data. Results of the review and validation processes will be reported to the EPA Project Manager. In 2009, a copy of the review and validation report will be provided to the ODEQ Project Sampling Coordinator.

D2. Validation and Verification Methods

All Field Forms and Sample Custody & Analysis Required Forms will be reviewed by the Field teams. Any discrepancies in the records will be reconciled with the appropriate associated field personnel and will be reported to the EPA Project Manager. In 2009, the validation report will be provided to the ODEQ Project Manager.

The submission of samples to the laboratory will include Sample Custody & Analysis Required Form documenting sampling time and date. This information will be checked by the receiving laboratory to ensure that holding times have not been exceeded. Violations of holding times will be reported (by the laboratory) to the USEPA Project Manager, and in 2009 to the ODEQ Project Manager.

For this project, in 2009, the ODEQ Project Manager will review all data to determine if the data quality objectives were met for each analytical batch. Trace level analyses are very susceptible to blank contamination. *The results from field and analytical blanks will not be censored unless the values are less than the LOD.* The ODEQ Project Sampling Coordinator will review lab-qualified data, flags and comments, and may adjust qualifier codes (e.g. Acceptable, Estimate, Questionable) to reflect the project's QAPP requirements.

D3. Reconciliation with Data Quality Objectives

As soon as possible following completion of the sample collection task, precision, accuracy, and completeness measures will be assessed by EPA / ODEQ and compared with the criteria shown in Tables 3 through 6. This will represent the final determination of whether the data collected are of the correct type, quantity, and quality to support their intended use for this project. Any problems encountered in meeting the performance criteria (or uncertainties and limitations in the use of the data) will be discussed with the EPA Project Manager, and will be reconciled, if possible. In 2009, the ODEQ Project Manager and CRITFC Project Manager will assess the data and discuss any problems with the EPA Project Manager.

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